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Quartz in Coal Dust Deposited on Internal Surface of Respirable Size Selective Samplers

Jhy-Charm Soo[1], **Taekhee Lee**[1], **Michael Kashon**[2], **Mohannad Kusti**[1], and **Martin Harper**[1]

[1]Exposure Assessment Branch, Health Effects Laboratory Division, National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention, Morgantown, West Virginia

[2]Biostatistics and Epidemiology Branch, Health Effects Laboratory Division, National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention, Morgantown, West Virginia

Abstract

The objective of the present study is to quantify quartz mass in coal dust deposited on the internal cassette surface of respirable size-selective samplers. Coal dust was collected with four different respirable size-selective samplers (10 mm Dorr-Oliver nylon [Sensidyne, St. Petersburg, Fla.], SKC Aluminum [SKC Inc., Eighty Four, Pa.], BGI4L [BGI USA Inc., Waltham, Mass.], and GK2.69 cyclones [BGI USA Inc.]) with two different cassette types (polystyrene and static-dissipative polypropylene cassettes). The coal dust was aerosolized in a calm air chamber by using a fluidized bed aerosol generator without neutralization under the assumption that the procedure is similar to field sampling conditions. The mass of coal dust was measured gravimetrically and quartz mass was determined by Fourier transform infrared spectroscopy according to the National Institute for Occupational Safety and Health (NIOSH) Manual of Analytical Methods, Method 7603. The mass fractions of the total quartz sample on the internal cassette surface are significantly different between polystyrene and static-dissipative cassettes for all cyclones ($p < 0.05$). No consistent relationship between quartz mass on cassette internal surface and coal dust filter mass was observed. The BGI4L cyclone showed a higher (but not significantly) and the GK2.69 cyclone showed a significantly lower ($p < 0.05$) internal surface deposit quartz mass fraction for polystyrene cassettes compared to other cyclones. This study confirms previous observations that the interior surface deposits in polystyrene cassettes attached to cyclone pre-selectors can be a substantial part of the sample, and therefore need to be included in any analysis for accurate exposure assessment. On the other hand, the research presented here supports the position that the internal surface deposits in static-dissipative cassettes used with size-selective cyclones are negligible and that it is only necessary to analyze the filter catch.

Address correspondence to: Taekhee Lee, 1095 Willowdale Road M/S 3030, Morgantown, West Virginia 26505; fwc8@cdc.gov.

INTRODUCTION

Impingers used in the early days of aerosol exposure assessment collected particles in a liquid suspension and the particles were then counted under a microscope where they could also be sized to determine health-related size fractions. Impingers, however, have disadvantages, and since the 1940s[(1)] and 1950s[(2)] filters have been used to collect particles in air for analysis of aerosol concentrations. Filters are more efficient than impingers at collecting very fine particles. Once occupational exposure limits were converted from number concentration to mass concentration in the 1960s, filters allowed more rapid analysis by the simple difference between pre- and post-sampling weights. It was necessary to include a device for size-separation prior to the filter when assessing the hazard from only fine particles.[(3)]

Conventions for particle penetration into the alveolar region of the human airways were established. Devices, notably miniature cyclones, such as the 10 mm Dorr-Oliver nylon cyclone, were developed to effect the size separation in accordance with these conventions.[(4)] Cyclones consist of an entry inlet, a body in which a vortex airflow effects size separation, and an outlet, often referred to as the “vortex finder,” directing the flow containing the smaller size fraction to the filter. This vortex finder is usually only a few millimeters in diameter, while the filter is much larger, nominally 25 mm or 37 mm diameter (the effective diameter for the filtration area being slightly smaller). Thus the filter is maintained in a holder at some distance above the outlet of the vortex finder. It has been typical for researchers to assume no loss of particles between the vortex finder and the filter, for example, on the internal surfaces (“walls”) of the filter holder, when conducting experiments in the analysis of cyclone performance. In fact, modern practice is often to connect the outlet of the vortex finder directly to a real-time aerosol measuring instrument, thereby bypassing any possibility of such losses.

An early indication that such losses might be important can be found in a paper presented at the ASTM International Symposium on Silica Sampling and Analysis held in Salt Lake City, Utah in 2004, and subsequently published in the *ASTM Journal*, and further reprinted in the Selected Technical Papers (proceedings) of the Second ASTM International Symposium on Respirable Crystalline Silica and Related Mineral Particles.[(5)] This work arose out of a concern that variability in any deposits on the internal surfaces of the filter holder compared to those on the filter of proficiency test samples for respirable silica prepared through aerosol generation might lead to high variability in the analysis when only the filter was analyzed. The paper demonstrated that a highly variable and potentially large proportion of the sampled particles could be found on the internal surfaces of both two-piece and three-piece polystyrene cassettes used with the Dorr-Oliver nylon cyclone. The researchers also examined field samples and noted the similar variability whether the cassettes were 25 mm or 37 mm diameter. The objective of the current work is to expand upon that study in another laboratory, using coal dust with a consistent quartz content, and extend it to include several other cyclones and types of cassettes, in support of recommendations to assess workplace aerosol concentrations most accurately.

METHOD

Coal Dust Sampling and Gravimetric Analysis

The respirable size-selective samplers that were employed in this study were: 1) 10-mm Dorr-Oliver nylon cyclone (Sensidyne, St. Petersburg, Fla.) operating at 1.7 L min^{-1}, 2) SKC Aluminum cyclone (SKC Inc., Eighty Four, Pa.) operating at 2.5 L min^{-1}, 3) BGI4L cyclone (Higgins-Dewell type, nickel-plated aluminum body and aluminum grit pot, BGI USA Inc., Waltham, Mass.) operating at 2.2 L min^{-1}, and 4) GK2.69 cyclone (BGI Inc., operating at 4.4 L min^{-1}). The samplers were placed in a calm air chamber which has been described in a previous study. [6] Test atmospheres were generated using finely ground Pittsburgh seam coal dusts and a fluidized bed aerosol generator (Model 3400, TSI Inc., Shoreview, Minn.) without neutralization under the assumption that the procedure is similar to field sampling conditions.[7] Information regarding the particle size distribution of these aerosols has been described previously.[6]

Polyvinyl chloride (PVC) filters of 25 and 37 mm (GLA-5000, SKC Inc.) diameter were equilibrated at least 72 hr in a weighing room which provides constant relative humidity and temperature. Filters were passed through an electrostatic bar (Mettler-Toledo, Columbus, Ohio) before weighing to dissipate static charge. Weighing was performed with a micro balance (XP6U, readability 0.1 μg, Mettler-Toledo) for PVC filters. Measurements were made after allowing exactly 180 seconds for balance stabilization and the coefficients of variation (CV) of PVC blank filters ranged from 0.03 to 0.06 %. The pre-weighed filters were assembled in two different cassettes i.e., polystyrene (225–2259; 25 mm 3-piece cassettes and 225-3250; 37 mm 3-piece cassettes, SKC Inc.) and static-dissipative polypropylene cassettes (225-309, SKC Inc.) and leak-checked using a cassette leak tester (225-8531, SKC Inc.). Three-piece cassettes for 10 mm Dorr-Oliver nylon cyclone and two-piece cassettes (bottom piece plus middle ring) for other cyclones were used for sampling.

Personal air sampling pumps (PCXR4 and PCXR8, SKC Inc.) were connected to the cyclones. The flow rates through the samplers were calibrated using a Gilibrator-2 calibration device (800271, Sensidyne), using a calibration jar (225-112, SKC Inc.) for the 10 mm Dorr-Oliver nylon cyclone. The calibration jar had been checked for leaks and sealed where necessary. The inlets of BGI4L (BGI USA Inc.) and GK2.69 (BGI USA Inc.) cyclones were connected directly to the Gilibrator-2. The flow rates were calibrated before and after sampling to ensure the flow rate did not change by more than ±5%.

Six cyclones, three with polystyrene and three with static-dissipative cassettes, were placed in the calm air chamber and coal mine dust was collected by all samplers over the same time period. Two different levels of mass were tested with at least three repetitions for each level. In addition, some tests were carried out using the 10 mm Dorr-Oliver nylon cyclone with three 37 mm polystyrene cassettes and three 25 mm polystyrene cassettes to determine if the size of cassette made a difference to the internal surface deposit. The mass concentration of coal dust was determined using coal dust mass, pre- and post-flow rate, and sampling time for each sample obtained after sampling. Sampling duration was varied depending on mass concentration level between 60 and 150 min. A total of 216 samples was collected.

Fourier Transform Infrared (FTIR) Analysis for Quartz Quantification

Quartz measurement followed the National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods (NMAM)* Method 7603[8] and the procedures have been described in detail in a previous study.[6] Briefly, each post-weighed filter was placed in a 30 mL porcelain crucible with lid (5013, Avogadro's Lab Supply Inc., Miller Place, N.Y.) and ashed in a muffle furnace (Model F6010, Barnstead/ThermoLyne, Dubuque, Iowa) for 2 hr at 600°C. Coal dust deposited inside of the cassettes was wiped out with two or three successive PVC filters wetted with isopropyl alcohol until the surface of the cassettes was clean to visual inspection. The wiped PVC filters were ashed as described previously. The ashed sample was redeposited on an acrylic copolymer membrane filter (DM450, 0.45 μm, 47 mm, PALL Life Sciences, Ann Arbor, Mich.) and the quartz mass was quantified by reading peak height at 800 cm^{-1} with baseline between 820 and 670 cm^{-1} using FTIR (Nicolet 6700, Thermo Fisher Scientific, Waltham, Mass.) with the Omnic software package (version 8.1.11, Thermo Fisher Scientific).

Data Analysis

Data were analyzed using SAS/STAT software, Version 9.3 of the SAS System for Windows (SAS Institute, Cary, N.C.). The three values from replicate samples from a given trial were averaged prior to analysis. Data were transformed using the natural log to meet the assumptions of the analysis. All variables were analyzed with Proc Mixed using a two-factor (cassette type by sampler type) analysis of variance (ANOVA). Post-hoc comparisons and interaction contrasts were carried out using the slice option in Proc Mixed and Fisher's Least Significant Difference (LSD).

RESULTS

Respirable Dust and Quartz Mass Concentration

The mass concentrations of respirable coal dust and quartz (net masses in parentheses) determined from the filters only of the polystyrene cassettes ranged from 0.623 to 16.1 mg m^{-3} (0.134 to 7.74 mg) and 0.095 to 1.55 mg m^{-3} (0.017 to 0.745 mg), respectively. Themass concentrations of respirable coal dust and quartz determined from the filters only of the static-dissipative cassettes ranged from 0.824 to 16.8 mg m^{-3} (0.084 to 9.03 mg) and 0.072 to 1.85 mg m^{-3} (0.010 to 0.818 mg), respectively.

Wall-Deposited Quartz Mass Fraction

Quartz masses between the limit of detection (LOD; 10 μg from *NMAM 7603*) and limit of quantification (LOQ, around 30 μg from *NMAM* 7603) (mostly internal cassette surface deposits from static-dissipative cassettes) were included in calculations. Internal cassette surface-deposited quartz mass fractions

$$\left(\frac{Quartz\ mass\ on\ cassette\ internal\ surface}{Quartz\ mass\ on\ filter+quartz\ on\ cassette\ internal\ surface} \times 100\right)$$

between two different cassettes collected with four different cyclones are shown in Figure 1. The mass fractions of the total quartz sample on the internal cassette surface are significantly different between polystyrene and static-dissipative cassettes for all cyclones ($p < 0.05$). No consistent

relationship between quartz mass on cassette internal surface and coal dust filter mass was observed. The BGI4L cyclone (BGI USA Inc.) showed larger (but not significantly) and the GK2.69 cyclone (BGI USA Inc.) showed significantly lower ($p < 0.05$) internal surface deposit quartz mass fraction for polystyrene cassettes compared to other cyclones (Table I Internal surface deposit quartz mass fraction between 25 mm (median 12%, maximum 33%) and 37 mm (median 8.7%, maximum 30%) polystyrene cassettes with 10 mm Dorr-Oliver nylon cyclones did not show significant differences ($p > 0.05$).

DISCUSSION

Comparison by Cassette Type

Figure 2 shows a picture of (a) coal dust on internal surfaces of a polystyrene cassette when the BGI4L cyclone (BGI USA Inc.) was exposed at 14.4 mg m^{-3} (4.70 mg coal dust mass on filter) and (b) the coal dust wiped from the internal surfaces of the same cassette, using PVC filters as wipes. The quartz mass in the wipes was 63.0 μg (from FTIR measurement), but it is notable that this sample, selected for illustrative purposes, would represent a respirable dust concentration three times higher than the American Conference of Governmental Industrial Hygienists (ACGIHR®) threshold limit value (TLVR®). It is well known that a significant fraction of airborne particles can be deposited on the inside of sampling cassettes for “total” or “inhalable” dust due to a combination of electrostatic, impaction and interception, and gravitational and diffusion mechanisms.[9–14] Likewise, airborne occupational particles sampled with a respirable size selective sampler can be deposited on the interior surface of sampling cassettes due to electrostatically charged filters, [15] electrostatically charged sampling cassettes, and the airflow pattern inside the cassette,[16,17] and this could be up to 55% of the aspirated sample.[5] A possible solution for this problem is to utilize air sampling cassettes composed of static-dissipative (graphite-loaded) polymer, although internal deposits cannot be eliminated entirely.[17,18] The present study produced results consistent with these previous studies, i.e., quartz concentrations on interior surfaces of polystyrene cassettes (up to 43%), that were significantly larger than those on static-dissipative cassettes.

Comparison by Cyclone Type

The GK2.69 cyclone (BGI USA Inc.) with a polystyrene cassette had significantly lower (about 2.5 times) interior surface-deposited quartz mass fractions compared to other cyclones ($p < 0.05$). But there was no significant difference between the interior surface-deposited mass fractions on polystyrene cassettes from the 10 mm Dorr-Oliver nylon (Sensidyne) nylon, SKC Aluminum, and BGI4L cyclones (BGI USA Inc.). No significant difference, however, was found in the quartz mass fraction deposited on the internal surface of static-dissipative cassettes between any cyclones. One possible explanation for the difference between cyclones with polystyrene cassettes might be the larger diameter of the vortex finder of the GK2.69 cyclone (BGI USA Inc.), compared to those of the other cyclones. The flow pattern in a related cyclone with a small diameter vortex finder was observed to reach the filter, move outwards to the wall of the cassette, and then downwards into a toroidal path.[17] Due to the larger size of vortex finder of the GK2.69 cyclone (BGI USA Inc.) and lower air velocity, the flows outwards to the wall and downwards into a

toroidal path are minimized and this is also the reason why the GK2.69 cyclone (BGI USA Inc.) produces a uniform particle distribution on the filter.[19]

CONCLUSION

This study confirms previous observations that the interior surface deposits in polystyrene cassettes attached to cyclone pre-selectors can be a substantial part of the sample, and therefore need to be included in any analysis for accurate exposure assessment. Polystyrene cassettes are more commonly used in current exposure measurements than are static-dissipative polypropylene cassettes, but the latter are only slightly more expensive. If the internal surface deposits of the static-dissipative cassettes are considered to be minimal in relation to the overall sample, then there would be no need to wipe the interiors of the cassettes, which is a considerable burden on the analyst, inevitably increasing the cost of the analysis. Therefore, it is possible that static-dissipative cassettes could be used in respirable aerosol sampling to eliminate the need for wiping internal surfaces.

Most of samples (95%) taken with any of the cyclones tested using a static-dissipative cassette have more than 90% of the dust passing through the cassette and reaching the filter. The small bias (<10%) of dust on the internal surface of these cassettes needs to be balanced against the increased variance from the additional step of wiping the interior to account for the internal deposits when deciding the most appropriate procedure for sampling and analysis. NIOSH has previously published a recommendation to use static-dissipative cassettes with size-selective cyclones (respirable and thoracic).[9] Internal sampler surface deposits were anticipated to be negligible and an additional step for the recovery of internal surface deposits therefore was not included in the recommendation. The research presented here supports the position that the internal surface deposits in static-dissipative cassettes used with size-selective cyclones are negligible and that it is only necessary to analyze the filter catch. However, the mechanisms for particle deposition on internal sampler surfaces vary with particle size and so this recommendation does not extend to sampling systems for larger particles. A correction factor could be applied to a large body of data to provide some comparison between data sets collected with polystyrene and static-dissipative cassettes, but the large variation in ratios of filter to internal surface deposits means that the correction factor could not be used to accurately correct individual sample results.

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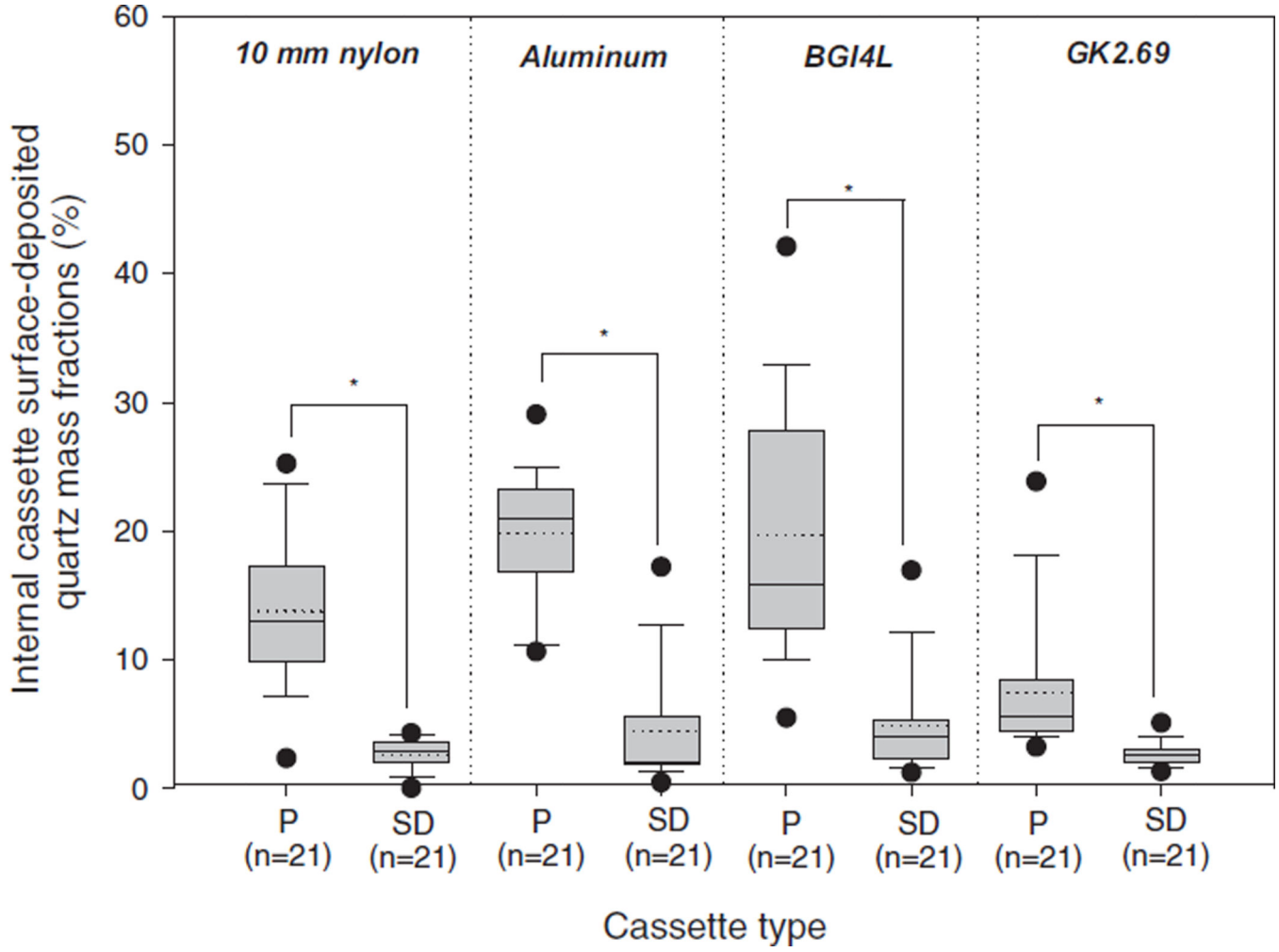

Figure 1.

Comparisons of internal cassette surface-deposited quartz mass fractions between two cassette types with 10 mm Dorr-Oliver nylon, Aluminum, BGI4L, and GK2.69 cyclones. The horizontal lines in the box plot from bottom to top indicate 10th, 25th, 50th (median), 75th, and 90th percentiles. The circles indicate the 5th (lower circle) and 95th (upper circle) percentiles. Dotted line indicates mean value. P is polystyrene cassette and SD is static-dissipative cassette. *N* is the number of samples for each sampler. * Statistically significant different ($p < 0.05$).

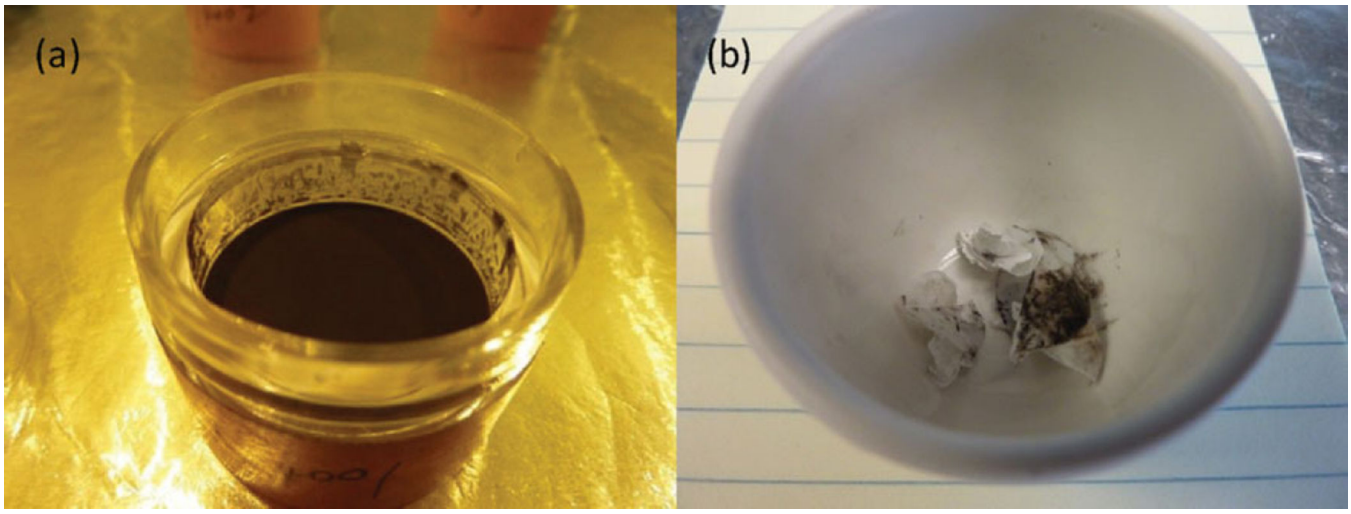

Figure 2.
Example picture of (a) coal dusts on internal surface of polystyrene cassettes and (b) wiped coal dust with PVC filters (placed in crucible). (color figure available online)

TABLE I

Internal Surface Deposit Quartz Mass Fraction (%) in Polystyrene and Static-Dissipative Cassettes Collected with Four Different Cyclones

Cassette type and size	Cyclone	Median (Mean)	75th percentile	90th percentile	Maximum
37 mm polystyrene	10 mm Dorr-Oliver nylon, SKC Aluminum, BGI4L	16.9 (17.9)	22.5	28.5	43.1
37 mm polystyrene	GK2.69 only	5.6 (7.52)	8.5	18.2	24.1
37 mm static-dissipative	10 mm Dorr-Oliver nylon, SKC Aluminum, BGI4L, GK2.69	2.80 (3.73)	4.30	6.74	17.6

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